

| | | |
|---|---|-------------|
| DRUG CHEMISTRY SECTION TECHNICAL PROCEDURE MANUAL | | |
| Procedure F-01 | Perkin Elmer Infrared Spectrophotometry | |
| Effective Date: | May 10, 2010 | Page 1 of 6 |

Name of Procedure:

Perkin-Elmer Infrared Spectrophotometry

Suggested Uses:

Collection of quantitative or qualitative data for the identification of controlled and non-controlled substances.

Apparatus Used to Perform Procedure:

Perkin-Elmer models: 1600, Paragon 1000, PE RXI, Spectrum BX, Spectrum One or Spectrum 100
 Perkin-Elmer Universal Attenuated Total Reflectance (ATR) Sampling Accessory
 Stainless steel vial and ball
 Vacuum pump and tubing
 Power conditioner
 Plotter/pens or printer/ink cartridge
 Paper
 Spatula
 Agate mortar and pestle
 Polystyrene film standard
 Polystyrene film standard spectra (attachment)
 Oven
 Thermometer

| | | |
|---|---|-------------|
| DRUG CHEMISTRY SECTION TECHNICAL PROCEDURE MANUAL | | |
| Procedure F-01 | Perkin Elmer Infrared Spectrophotometry | |
| Effective Date: | May 10, 2010 | Page 2 of 6 |

Performance Verification Requirements:

Each IR instrument shall have a monthly performance verification to ensure proper functioning.

The internal polystyrene of each IR instrument shall be compared to a traceable standard yearly.

Performance Verification procedure:

Monthly:

1. Double click on the "spectrum software" icon
Log-in and click through the prompts.
2. Scan background
Click on "scan and instrument set-up" hotkey
("scan" tab-check for Scan duration set to 4)
("Instrument" tab-check for Resolution se to 4.00 cm⁻¹)
"Background" hotkey
Scan
3. Scan using internal polystyrene
Click on "Scan and Instrument Setup" hotkey
"Beam" tab
Double click on the picture with disk/holes on the left-hand side
Choose "polystyrene"
OK
Apply
Scan
4. Click "Label peaks" hotkey
Print the scan with at least the following three wave numbers marked by the Label Peaks software:

2849 cm⁻¹, 1942 cm⁻¹, and 906 cm⁻¹ (+/- 3cm⁻¹).
5. Reset the beam and instrument for casework samples:
Click on "Scan and Instrument Setup" hotkey
"Beam" tab
Double click on picture with disk/holes on the left
Choose "None"
OK (This removes the polystyrene from the beam.)

F – 01

| | | |
|---|---|-------------|
| DRUG CHEMISTRY SECTION TECHNICAL PROCEDURE MANUAL | | |
| Procedure F-01 | Perkin Elmer Infrared Spectrophotometry | |
| Effective Date: | May 10, 2010 | Page 3 of 6 |

Apply
 "Contamination check" hotkey
 OK (This runs a contamination check in preparation for samples.)

6. The allowable variance from the certified value for the peaks appearing at these wave numbers is $\pm 0.5 \text{ cm}^{-1}$. If the results are outside these specifications, the instrument will be removed from casework immediately and the following shall be done:
 - a. Place an "Out of Service" sign on the front of the instrument.
 - b. Notify the IR Coordinator so he/she can call the Service Engineer to schedule an on-site assessment.
7. The monthly polystyrene scan will be filed and maintained by the IR Coordinator in a notebook that is kept with each instrument.

Yearly:

Internal Polystyrene Verification:

A scan of a certified, traceable polystyrene film shall be collected with the KBr accessory in place, followed by the collection of a scan of the internal polystyrene with the ATR attachment in place, according to the procedure above. The data obtained shall be evaluated using the criteria in the procedure above. This data will be filed for each instrument and maintained by the IR Coordinator in the Section Conference Room.

New Instrument Set Up:

1. New FTIR instruments will be installed by a Perkin-Elmer engineer according to Perkin-Elmer policy.
2. Upon completion of the installation a scan of a certified, traceable polystyrene film shall be collected with the KBr accessory in place, followed by the collection of a scan of the internal polystyrene with the ATR attachment in place, according to the procedure above. The data obtained shall be evaluated using the criteria in the procedure above. This data will be filed for each instrument and maintained by the IR Coordinator in the Section Conference Room.

F – 01

Page 3 of 6

This document is not controlled if printed.

| | | |
|---|---|-------------|
| DRUG CHEMISTRY SECTION TECHNICAL PROCEDURE MANUAL | | |
| Procedure F-01 | Perkin Elmer Infrared Spectrophotometry | |
| Effective Date: | May 10, 2010 | Page 4 of 6 |

3. Scans from at least three controlled substances will also be obtained, following polystyrene confirmation, at instrument setup. Examples include: Methamphetamine, Phentermine, and Cocaine Base, but other controlled substances may be used depending on availability of standards. The data obtained shall be reviewed by the IR Coordinator and found to be substantially the same as the library standard for that compound. This data will be filed for each instrument and maintained by the IR Coordinator in the Section Conference Room.

Application of Procedure For Solid Samples Using the ATR:

1. Clean the attenuated total reflectance (ATR) sampling accessory crystal using water or an organic solvent. Ensure that the crystal is completely dry.
2. Perform a contamination check to ensure that the crystal has been properly cleaned. If the contamination check fails, repeat the cleaning procedure.
3. Place approximately 1 milligram of sample evenly onto the ATR crystal.
4. Apply appropriate force using the ATR force arm to ensure good contact between the sample and the surface of the crystal.
5. Scan to acquire data.
6. Data can now be processed in any number of ways including: flattened, abexed, and rescaled.
7. Print completed scan and compare to a known reference standard.

Application of Procedure For Liquid Samples Using the ATR:

1. Clean the attenuated total reflectance (ATR) sampling accessory crystal using water

| | | |
|---|---|-------------|
| DRUG CHEMISTRY SECTION TECHNICAL PROCEDURE MANUAL | | |
| Procedure F-01 | Perkin Elmer Infrared Spectrophotometry | |
| Effective Date: | May 10, 2010 | Page 5 of 6 |

or an organic solvent. Ensure that the crystal is completely dry.

2. Perform a contamination check to ensure that the crystal has been properly cleaned. If the contamination check fails, repeat the cleaning procedure.
3. Apply enough of the liquid sample to cover the ATR crystal
4. Scan to acquire data.
5. Data can now be processed in any number of ways including: flattened, abexed, and rescaled.
6. Print completed scan and compare to a known reference standard.

Safety Concerns:

Do not look directly into the laser beam emitted from the infrared spectrophotometer.
Do not over tighten the force gauge.
Avoid contact with the organic solvents near the eyes.

Literature References:

Moffat, A. C. Ed., **Clarke's Isolation and Identification of Drugs**, 2nd. Ed., The Pharmaceutical Press, 1986.

Mills, III, Terry and Roberson, Conrad J., **Instrumental Data for Drug Analysis**, 2nd Ed., Vols. 1-5, CRC Press, Inc., 1993.

Silverstein, R. M. And Brassler, Clayton G., and Terence C. Morrill, **Spectrometric Identification of Organic Compounds**, New York, Wiley, 1991.

Keller, Roger, **The Sigma Library of FT-IR Spectra**, Edition 1, Vol. 1 and 2, Sigma Chemical Company, Inc., 1986.

Pouchert, Charles J., **The Aldrich Library of Infrared Spectra**, Aldrich Chemical Company, 1981.

| | | |
|---|---|-------------|
| DRUG CHEMISTRY SECTION TECHNICAL PROCEDURE MANUAL | | |
| Procedure F-01 | Perkin Elmer Infrared Spectrophotometry | |
| Effective Date: | May 10, 2010 | Page 6 of 6 |

UNCONTROLLED DOCUMENT IF PRINTED